Characterization of Cryogenically-Cycled Autoclaved and ATL CF/PEEK Laminates using 3-D X-Ray CT

CHARACTERISATION OF CRYOGENICALLY CYCLED AUTOCLAVE & ATL CF/PEEK LAMINATES USING 3-D X-RAY CT

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ABSTRACT

Non-destructive testing (NDT) techniques including optical microscopy and 3-D X-ray computed tomography (CT) are used to characterise and compare the defect content and damage formation in cryogenically cycled CF/PEEK specimens manufactured using two distinct processing methods: autoclave and automated tape laying (ATL). Significant differences in void volume content as well as transverse microcrack density are observed in the specimens post-cycling, with the presence of large air gaps in the ATL specimens likely contributing to the observed damage accumulation behaviour.

1 INTRODUCTION

Due to their high specific strength and stiffness amongst other properties, carbon-fibre reinforced polymers (CFRP) are seen as candidate materials for the fuel tanks of next generation reusable launch vehicles (RLVs). These fuel tanks will be exposed to cryogenic temperatures as low as -250 °C, leading to microcracking and delamination formation within the CFRP, which, in severe cases, can result in permeation of the cryogen through the fuel tank walls. A precise understanding, therefore, of the material structure and damage accumulation underpins the potential use of CFRP for RLVs [1-3]. This work investigates and compares the cryogenic performance of CF/PEEK, a high-performance thermoplastic CFRP, processed using two distinct manufacturing methods: autoclave and automated tape laying (ATL), using advanced non-destructive testing (NDT) techniques.

2 METHODOLOGY

2.1 Materials

Two sets of CF/PEEK specimens were manufactured from Suprem T/60%/IM7/PEEK/150 [4] using autoclave and ATL processing techniques. The autoclave specimens, manufactured at ÉireComposites Teo, were processed at a temperature of 375 °C and pressure of 6.5 bar, with an average cooling rate of 3.8 °C [2]. The ATL specimens were processed using a KUKA KR 180 R2900 robot with a laser-line diode laser module (LDM) 3000W system operated by the Irish Centre for Composites Research (ICOMP). The process parameters included a lay-down speed of 6 m/min, a target temperature of 420 °C, a tool temperature of 280 °C, an average laser power of 500 W and a roller pressure of 4.5 bar [5]. A quasi-isotropic lay-up was used for both sets of specimens, although the stacking sequence and overall ply count differed slightly. The autoclave specimens were 16-ply $[0°_2/45°_2/135°_2/90°_2]_S$ laminates and the ATL specimens were 18-ply $[45°/135°/90°/0°/90°/0°/90°/0°/90°]_S$ laminates. Both sets of specimens were cut to rectangular coupons measuring 34 mm × 27 mm as shown in Fig. 1.

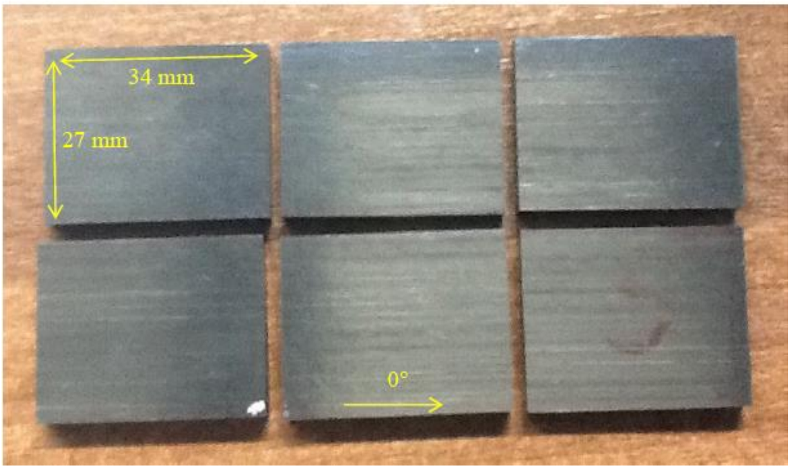

Figure 1: Rectangular CF/PEEK coupons used in testing.

2.2 Cryogenic testing

The cryogenic cycling of the specimens involved direct immersion in liquid nitrogen at a temperature of -196 °C before heating in a warm air flow at 40 °C, as shown in Fig. 2. The cycle dwell times necessary for the complete cooling and heating of the specimens were based on the temperature profile of a thermocouple embedded within the centre of a specimen subjected to a cryogenic cycle.

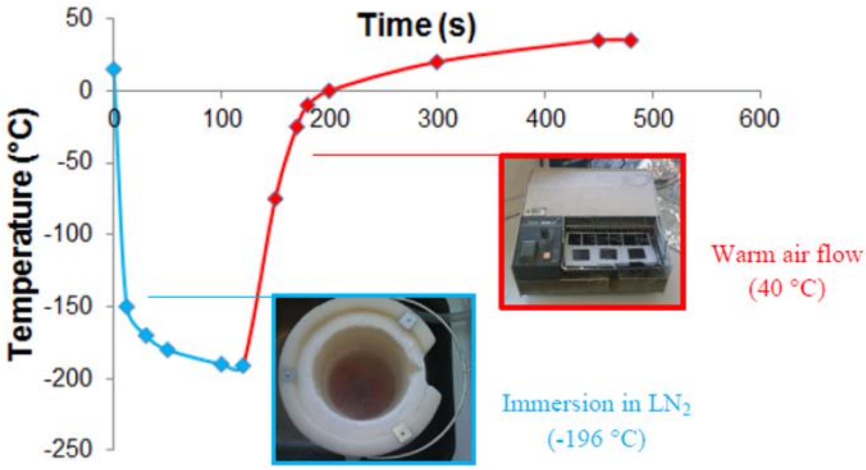

Figure 2: The cryogenic cycle which both the autoclave and ATL specimens were exposed to.

2.3 Material characterisation

General material and damage characterisation was carried out both before and after cryogenic cycling using optical microscopy and 3-D X-ray CT. Specimen preparation for optical microscopy involved the grinding of specimen sides using progressively finer grit paper ranging from P180 to P2400, before being machine polished on cloth using diamond solutions ranging from 6 µm to 0.25 µm. The CT scans were carried out at the European Space Research and Technology Centre (ESTEC) using a Phoenix M nano/microtom [6]. The X-ray gun was rated at 180 kV, with scans being carried out at 160 kV and 28 µA, giving a scan power of 4.5 W. The scan resolution was 33 µm. The tomographical reconstruction was carried out using Davos software and volume rendering with *VGStudio MAX 2.2*.

3 RESULTS

3.1 Defect characterisation

3-D X-ray CT scans of the specimens were conducted before cryogenic cycling in order to inspect the laminates for manufacturing defects such as voids and inclusions. An increased presence of such defects can result in the degradation of key material properties and are generally undesirable. The void and inclusion volume for both the autoclave and ATL laminates are shown in Table 1, with Fig. 3 showing the rendered scans for both laminates. The void content in the ATL specimens was found to be significantly higher than the autoclave specimens; however both were below the general standard of 1.5% set for aerospace components. The metallic inclusion content was found to be negligible for both specimens. Of greater concern for the ATL specimen was the presence of air gaps resulting from the tape laying process. These gaps likely have significant implications for the laminate properties and would act as gas leak paths if they align through the specimen thickness.

Specimen	Defect	Volume (%)
16-ply a/c	Void	0.17
16-ply a/c	Inclusion	< 0.01
18-ply ATL	Void	0.69
18-ply ATL	Inclusion	<0.01

Table 1: Void and inclusion volume content for the autoclave (a/c) and ATL specimens measured using 3-D X-ray CT

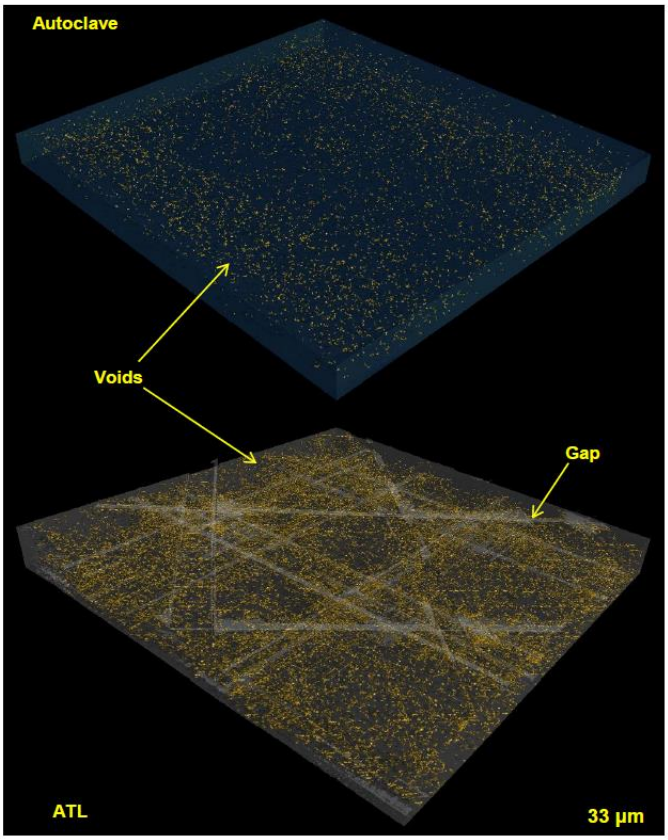

Figure 3: 3-D X-ray CT scans of voids in the autoclave (top) and voids and gaps in the ATL (bottom) specimens.

3.2 Damage characterisation

The specimens were inspected for damage after exposure to a single cryogenic cycle using both optical microscopy (Fig. 4) and 3-D X-ray CT (Fig. 5). Transverse microcracking was observed in both specimens post-cycling, although to a far greater extent in the autoclave specimen. The transverse microcracks and gaps were found to extend across the entire specimen width and through the thickness of each damaged ply.

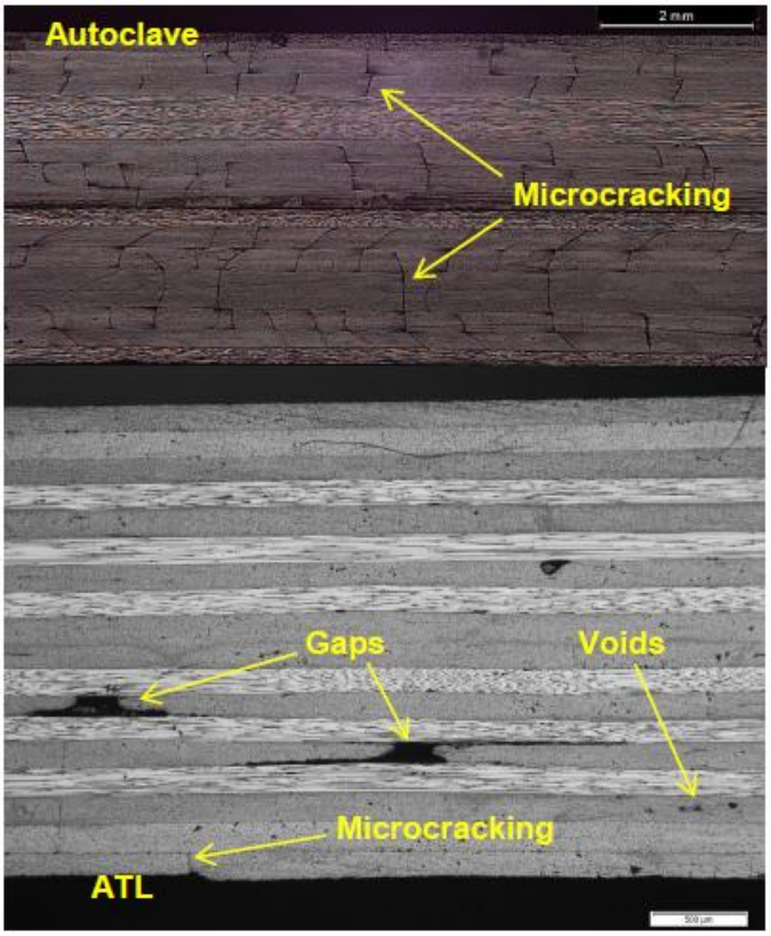

Figure 4: Optical micrograph of transverse micocracking in two autoclave specimens (top) and of microcracking and gaps in an ATL specimen (bottom) after exposure to a single cryogenic cycle.

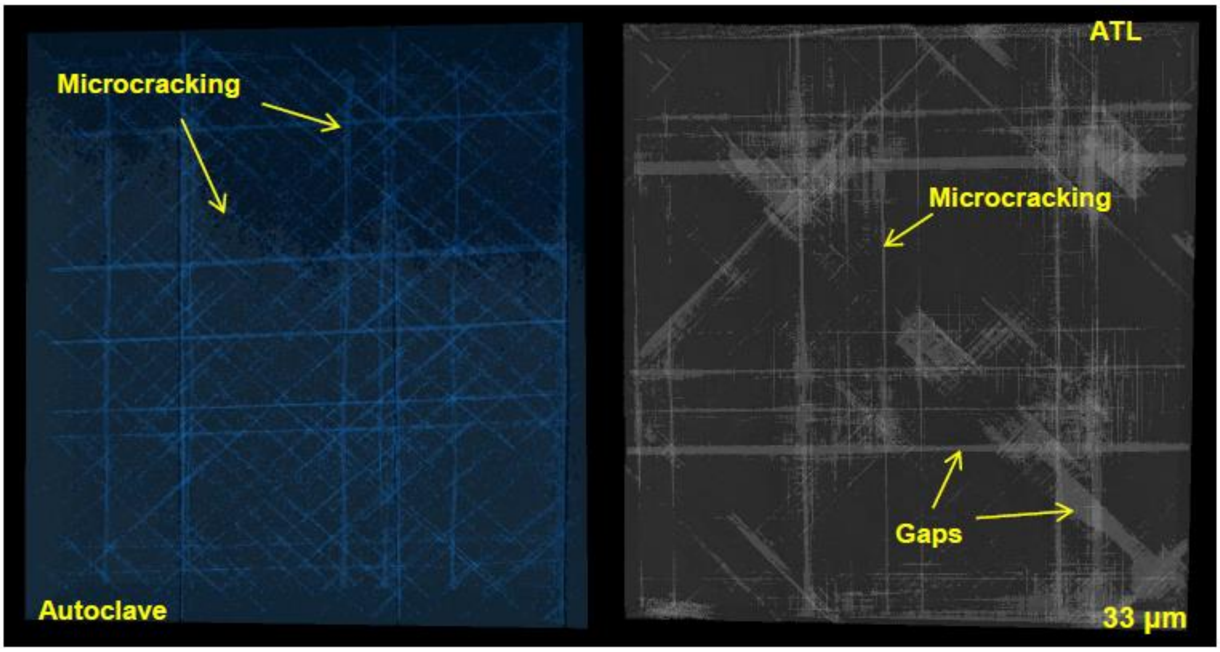

Figure 5: 3-D X-ray CT scans of microcracking in an autoclave specimen (left) and of microcracking and gaps in an ATL specimen (right) after exposure to a single cryogenic cycle.

In order to directly compare the damage formation in the autoclave and ATL specimens, the crack density for each ply was calculated by counting the number of transverse microcracks present on each specimen side from the optical micrographs. Fig. 6 shows the measured microcrack and microcrack/gap density for both specimens. Significantly more microcracking was observed in the 16-ply autoclave specimen despite the laminate containing less manufacturing defects and being of a higher general quality. Aside from the difference in laminate stacking sequence, the gaps in the 18-ply ATL specimen likely contribute to this difference in damage formation.

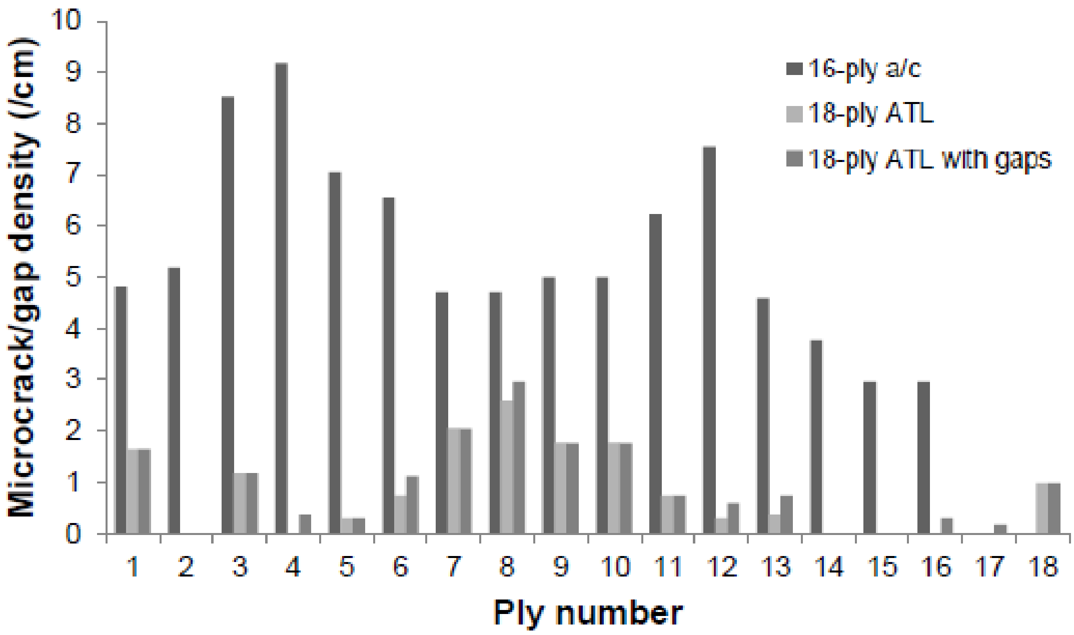

Figure 6: Chart of the microcrack density and microcrack/gap density for each ply of the autoclave (a/c) and ATL specimens after a single cryogenic cycle.

Although there are relatively few gaps in the ATL specimen, the gaps tend to have far larger opening displacements when compared to the microcracks. Fig. 7 compares the average crack opening displacements (COD) for the autoclave and ATL specimens without and with gaps included. The opening displacement of the gaps are on average several orders of magnitude greater than the transverse microcracks and represent major discontinuities in the material structure with implications for the thermal response and resulting stress build-up in the laminate. This can explain the reduced microcrack density in the ATL laminates. However despite having a lower microcrack density, the large gaps in ATL specimen make it susceptible to gas leakage, in cases where the microcracks and gaps overlap and connect through the laminate thickness.

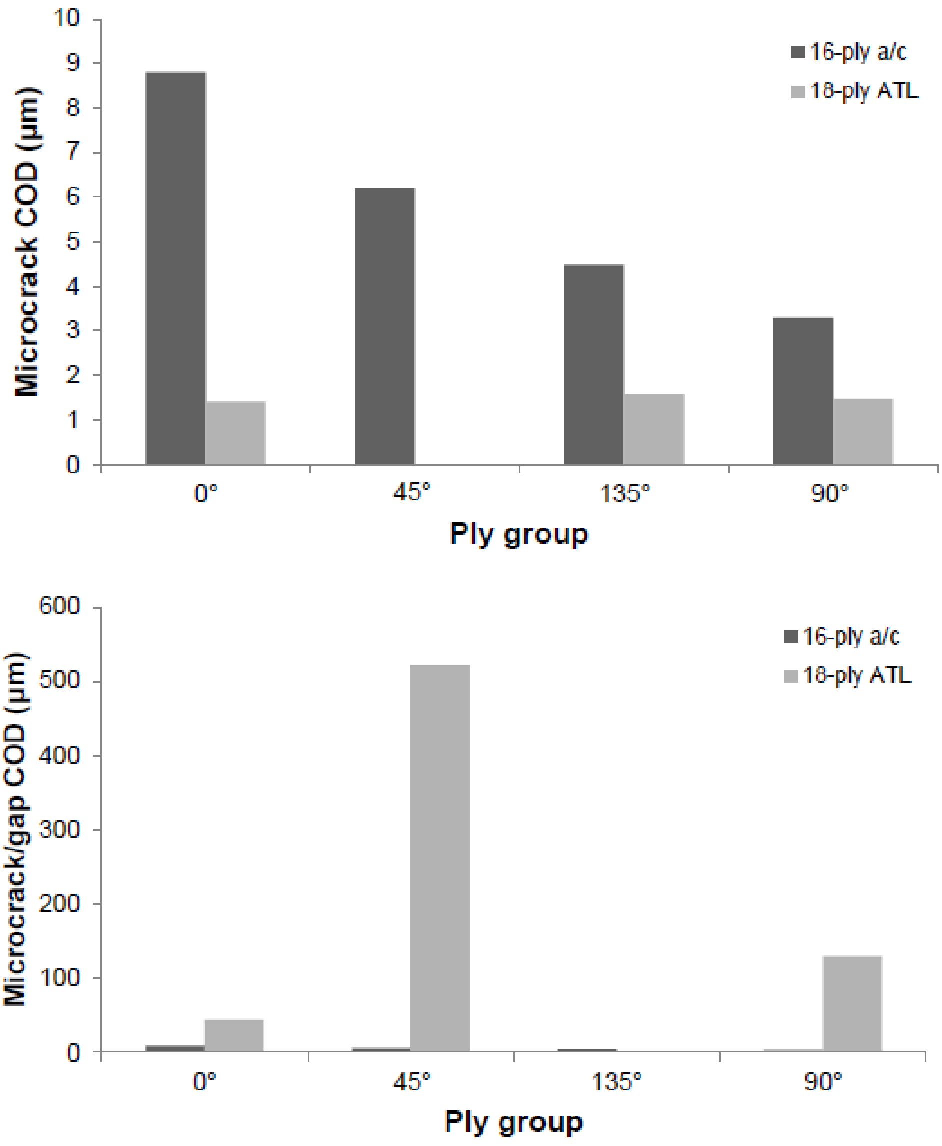

Figure 7: Comparison of the crack opening displacement (COD) of microcracks present in cryogenically cycled autoclave (a/c) and ATL specimens (top) and of the combined microcrack/gap opening displacements (bottom).

5 CONCLUSIONS

This work has presented a comparison of the defect contents and damage accumulation of CF/PEEK specimens manufactured using autoclave and ATL processing methods after exposure to cryogenic temperatures. Using 3-D X-ray CT, the void content of the ATL specimen was found to be significantly higher than the autoclave specimen, even without the inclusion of the large air gaps present in the ATL specimen due to processing. Optical microscopy and 3-D X-ray CT were used to observe and measure damage formation in the form of transverse microcracking. Noticeably higher crack densities were observed in the autoclave specimens, despite the overall higher quality of these laminates. However, the presence of several large gaps through the thickness of the ATL specimens greatly increases the likelihood of gas leakage, despite the relatively minor damage formation, with implications for cryogenic fuel tank design.

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